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Jawaharlal Nehru

“Step Out From the Old to the New”

IS 13630-10 (2006): Ceramic Tiles - Methods of test, Sampling and Basis of Acceptance, Part 10: Determination of frost resistance (see IS 13630 : Parts 1 to 15) [CED 5: Flooring, Wall Finishing and Roofing]



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“ज्ञान एक ऐसा खजाना है जो कभी चुराया नहीं जा सकता है”

Bhartrhari—Nitiśatakam

“Knowledge is such a treasure which cannot be stolen”



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IS 13630 (Part10) : 2006

भारतीय मानक  
सिरैमिक टाईलें - परीक्षण पद्धतियाँ,  
नमूने लेने तथा स्वीकार्यता का आधार  
( पहला पुनरीक्षण )

*Indian Standard*

CERAMIC TILES — METHODS OF TEST,  
SAMPLING AND BASIS FOR ACCEPTANCE  
( *First Revision* )

ICS 91.100.23

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## FOREWORD

This Indian Standard (Parts 1 to 15) (First Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Flooring, Wall Finishing and Roofing Sectional Committee had been approved by the Civil Engineering Division Council.

This standard was first published in various parts in 1992-93. This is the first revision; having all parts combined in one publication, of the standard in which the following major changes have been incorporated:

- a) As per the decision taken in the last meeting, the requirements of all the parts have been included in one volume and the revised standard has been brought in line with ISO 10545 (various parts).
- b) The requirements for determination of bulk density have been added in Part 2 and a few changes have been made in the requirements for determination of water absorption.
- c) A few modifications have also been made in Part 3.
- d) Requirements for determination of breaking strength have also been added in Part 6.
- e) Requirements for determination of glazing resistance tests have also been modified in Part 9.
- f) A new test for determination of impact resistance by measurement of co-efficient of restitution has been added as Part 14.
- g) IS 13711 : 1993 'Sampling and basis for acceptance' has been amalgamated with Part 15 of this standard.

In formulation of this standard considerable assistance have been derived from the following standards:

- ISO 10545-1 : 1995 Ceramic tiles — Part 1 : Sampling and basis for acceptance
- ISO 10545-2 : 1995 Ceramic tiles — Part 2 : Determination of dimensions and surface quality
- ISO 10545-3 : 1995 Ceramic tiles — Part 3 : Determination of water absorption, apparent porosity, apparent relative density and bulk density
- ISO 10545-4 : 2004 Ceramic tiles — Part 4 : Determination of modulus of rupture and breaking strength
- ISO 10545-5 : 1996 Ceramic tiles — Part 5 : Determination of impact resistance by measurement of coefficient of restitution
- ISO 10545-6 : 1995 Ceramic tiles — Part 6 : Determination of resistance to deep abrasion for unglazed tiles
- ISO 10545-7 : 1996 Ceramic tiles — Part 7 : Determination of resistance to surface abrasion for glazed tiles
- ISO 10545-8 : 1994 Ceramic tiles — Part 8 : Determination of linear thermal expansion
- ISO 10545-9 : 2004 Ceramic tiles — Part 9 : Determination of resistance to thermal shock
- ISO 10545-10 : 1995 Ceramic tiles — Part 10 : Determination of moisture expansion
- ISO 10545-11 : 1994 Ceramic tiles — Part 11 : Determination of crazing resistance for glazed tiles
- ISO 10545-12 : 1995 Ceramic tiles — Part 12 : Determination of frost resistance
- ISO 10545-13 : 1995 Ceramic tiles — Part 13 : Determination of chemical resistance
- ISO 10545-14 : 1995 Ceramic tiles — Part 14 : Determination of resistance to stains
- ISO 13006 : 1998 Ceramic tiles — Definitions, classification, characteristics and marking

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

## *Indian Standard*

# CERAMIC TILES — METHODS OF TEST, SAMPLING AND BASIS FOR ACCEPTANCE

## PART 10 DETERMINATION OF FROST RESISTANCE

### *( First Revision )*

#### 1 SCOPE

This standard (Part 10) covers a method of test for evaluating the frost resistance of all ceramic tiles intended for use in conditions of frost in the presence of water.

#### 2 PRINCIPLE

After impregnation with water tiles are cycled between +15° and -15°C. All sides of tiles are exposed to freezing during 50 freeze-thaw cycles.

#### 3 APPARATUS

3.1 **Drying Oven**, capable of operation at  $110 \pm 5^\circ\text{C}$ .

3.2 **Balance**, weighing to 0.1 percent of the mass of a test specimen.

3.3 **Container**, for impregnation by capillarity or immersion with stable horizontal supports.

3.4 **Apparatus for impregnation with water after evacuation** by means of a vacuum pump capable of lowering than air pressure by  $40 \pm 2.6$  kPa in a tank containing tiles.

3.5 A freezer capable of freezing at least ten tiles having a minimum total area of  $0.25\text{ m}^2$  supported in such a manner that the tiles shall be separated. The freezer shall be capable of reducing the temperature to  $-15 \pm 3^\circ\text{C}$  in 2 h, if the freezer is automated it shall comply with the freeze-thaw cycling conditions. The temperature shall be as uniform as possible in the freezer and it shall have air circulation (measured in the empty freezer) with a maximum air velocity of 4 m/s.

#### 3.6 Chamois Leather

3.7 Demineralized water, if water hardness less than 400 mg/l  $\text{CaCO}_3 + \text{MgCO}_3$  (or equivalent hard water salts) at 15° to 25°C is not available.

#### 4 TEST SPECIMENS

##### 4.1 Sample

A minimum area of  $0.25\text{ m}^2$  and not less than 10 whole tiles shall be used. The tiles shall be free from dust.

NOTE — If tiles with defects such as cracks, crazing, holes, nipped edges and nipped corners have to be tested then the defects shall be marked with a permanent stain prior to the test and these defects shall be examined after testing.

#### 4.2 Preparation

Dry the tiles in the oven at  $110 \pm 5^\circ\text{C}$  until constant mass is reached, that is, when the difference between two successive weighing at intervals of 24 h is less than 0.1 percent. Record the dry mass of each tile ( $M_1$ ).

#### 5 IMPREGNATION WITH WATER

##### 5.1 Progressive Water Impregnation by Capillarity Whatever the Water Absorption

5.1.1 Place the tiles vertically in the container with no contact between them and no contact with the container so that one lone side shall be standing on the supports.

5.1.2 Add water to the level of one-fifth of the height of the tiles. Add additional quantities of water at subsequent 24 h intervals so that a further one-fifth of the height shall be immersed each 24 h with the fifth and final addition of water add sufficient to immerse the tiles under a 50 mm head of water. After the fifth day allow the tiles to remain under water for a further period of 2 days so that they shall be fully immersed for 3 days.

5.1.3 Prepare chamois leather by wetting and wringing out by hand. Place the chamois leather on a flat surface and lightly dry each side of each tile in turn. Dab relief surface with chamois leather.

5.1.4 Weigh each tile immediately after this procedure.

5.1.5 Record the wet mass of each tile ( $M_2$ ).

##### 5.2 Water Impregnation at Atmospheric Pressure for Tiles with Water Absorption $\leq 6$ Percent

5.2.1 Place the tiles vertically in the container with no contact between them and no contact with the container so that one lone side shall be standing on the supports. Add water so as to completely immerse the tiles and allow to stand for 24 h.

5.2.2 Lightly dry the surface of each tile with damp chamois leather using procedure given in 5.1. Weigh

each tile. Repeat this procedure of natural immersion for further periods of 24 h each until the change in mass between two successive weighing shall be less than 0.1 percent of total mass. There shall be a minimum time of immersion of 3 days.

5.2.3 Record the wet mass of each tile ( $M_2$ ).

**5.3 Water Impregnation by Evacuation for Tiles with Water Absorption > 6 Percent**

5.3.1 Place the tiles vertically in the dry vacuum tank with no contact between them and no contact with the vacuum tank. Connect to the vacuum pump and evacuate to a pressure of  $40 \pm 2.6$  kPa below atmospheric pressure and maintain the pressure for 30 min. bring the water into the tank containing the tiles whilst maintaining this pressure and cover the tiles by at least 50 mm head of water. This operation shall take 30 min maintain the evacuation at the same pressure for a further 30 min and then restore atmospheric pressure. Leave the tiles under water for 1 h before removing them.

5.3.2 Lightly dry the surface of each tile with damp chamois leather using the procedure given in 5.1.

5.3.3 Record the wet mass of each tile.

**5.4 Calculation of Water Content**

The water content expressed as a percentage (by mass) is determined from:

$$W_{c_1} = \frac{(M_2 - M_1)}{M_1} \times 100$$

where

$W_{c_1}$  = water content by mass (percent),

$M_1$  = dry mass of each tile, and

$M_2$  = wet mass of each tile.

**6 PROCEDURE**

6.1 Select the appropriate impregnation method. The reference method to be used for arbitration shall be the method of progressive water impregnation by capillarity.

6.2 Select one of the thickest tiles that are under test. It shall be one considered to have repressive properties of that test sample. Drill a 3 mm diameter hole from the centre of one edge to a maximum distance of 40 mm from the edge of the tile. Insert thermocouple or other suitable measuring device and seal the hole with a tiny piece of thermally insulating material (for example, extended polystyrene). If it is not possible to drill a hole in this way, place a thermocouple on the center of each face of a tile and adhere a second tile over this.

6.3 Place all the tiles under test in the freezer in such way that there is air space between each one and air flow over all surfaces. Place the tile with thermocouple in the middle of the test samples. The temperature of the thermocouple defines the temperature of the entire tile under test. Only in the case of repeated tests with similar test specimens can this be omitted and then occasional check shall be made with a thermocouple in a tile. When there is no temperature reference tile the air temperature shall be measured. All temperature measurements shall be accurate to  $\pm 0.5^\circ\text{C}$ . Ideally testing shall then be proceeded by automated freeze-thaw cycling. Whether automated or not the temperature time relationship shall be within the range defined in Fig. 1.

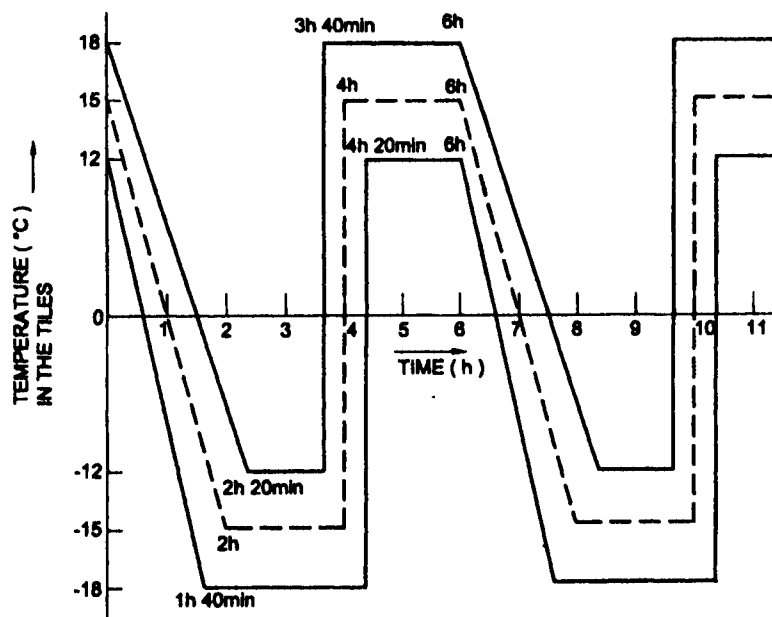


FIG. 1 SCHEMATIC REPRESENTATION OF TEMPERATURE CURVE

The freezer can be controlled by means of the air temperature but Fig. 1 refers to the temperature in the tiles under test. The ideal freeze-thaw cycle in the tile is indicated by dotted line and actual freeze-thaw cycle shall be within the zone delineated by the two other line. The cycle shall be completed after 6 h.

6.4 Immerse the tiles in water at  $+15 \pm 3^\circ\text{C}$  drain the water before reaching  $5^\circ\text{C}$ . The temperature shall be reduced to  $-15 \pm 3^\circ\text{C}$  in  $2\text{h} \pm 20\text{ min}$  from commencement of the cycling. Adequate provision shall be made to prevent evaporation of water during cooling if the water is drained at a temperature above  $5^\circ\text{C}$  keep the tiles at  $15 \pm 3^\circ\text{C}$  for  $2\text{h} \pm 20\text{ min}$  and then introduce water at  $+15 \pm 3^\circ\text{C}$  and maintain this for  $2\text{h} \pm 20\text{ min}$  repeat the cycling procedure 50 times, as shown in Fig. 2 weigh the tiles after test ( $M_3$ ) and then dry them to constant mass ( $M_4$ ). The final water content expressed as a percentage (by mass) is determined from:

$$W_{c_2} = \frac{(M_3 - M_4)}{M_4} \times 100$$

where

- $W_{c_2}$  = final water content by mass (percent),
- $M_3$  = wet mass of each tile after the test, and
- $M_4$  = dry mass of each tile after the test.

6.5 Examine the glazed or proper surfaces and the edges of the tiles by the naked eye, with the aid of spectacle if usually worn, after 50 cycles. They may be examined at the intermediate stages to expedite the reporting if there is a reason to believe that they might sustain damage relatively early in the test. Record all damage that is observed on the glazed or proper surfaces and the edges of the tiles.

7 TEST REPORT

The test report shall contain the following:

- a) Identification of the tile;
- b) Number of tiles in the test sample;
- c) Method used to impregnate the tiles with water;
- d) Water content obtained before the freeze-thaw test;
- e) Water content obtained after the freeze-thaw test;
- f) Description of defects before the test and all damage on the glazed or proper surfaces and the edges of the tiles after the freeze-thaw test; and
- g) Number of damaged tiles after 50 cycles.

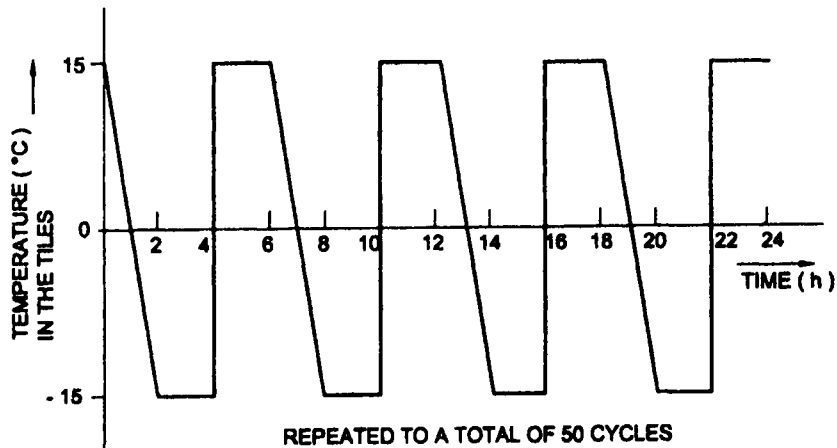


FIG. 2 SCHEMATIC DIAGRAM OF THE FREEZE-THAW CYCLES



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This Indian Standard has been developed from Doc: No. CED 5 (7358 to 7372).

### Amendments Issued Since Publication

Amend No.	Date of Issue	Text Affected

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